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# Magnetoimpedance of thin film meander with composite coating layer containing Ni nanoparticles

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Giant magnetoimpedance (GMI) [FeNi/Cu]<sub>4</sub>/FeNi/Cu/[FeNi/Cu]<sub>4</sub>/FeNi sensing meander elements were designed and tested aiming to develop a new instrumentation for characterization of polymer/magnetic nanoparticles composites. Acrylic copolymer of 95% of butyl methacrylate and 5% of methacrylic acid was used as polymer matrix of the composite. It was shown that GMI meander sensing elements are capable of detecting Ni nanoparticles spread in the polymer matrix. Polymeric composites filled with magnetic nanoparticles in small concentration are able to enhance GMI meander sensitivity. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4865319>]

Present day electronic and biomedical applications require the development of new types of functional composites. Among them one can mention polymer matrices (P) with magnetic nanoparticles (MNPs). Complete characterization of such complex nanomaterials becomes a strict requirement of their applications, especially in terms close to working conditions of the electronic components. Furthermore, P/MNPs composites could also be used to improve the sensitivity of thin film magnetic sensors. There are many techniques to fabricate large amount of the MNPs. One of them is the electric explosion of wire (EEW) method.<sup>1</sup>

Giant magnetoimpedance (GMI) is a large change of the high frequency impedance in certain soft ferromagnetic materials when constant magnetic field is applied.<sup>3</sup> When an alternating current flows through the ferromagnetic material, the effective cross section is reduced due to the classic skin effect.<sup>2</sup> GMI is interesting for low-magnetic field sensing, including magnetic biosensors.<sup>2-4</sup> In many cases, the surface of magnetic sensitive element should be protected in order to avoid corrosion, or to provide functionalization. On one hand, P/MNPs coverings of GMI sensitive element can be viewed as candidate to enhance the GMI and play a protective role at a time. On the other hand, GMI sensitive elements are good candidates to develop new types of instrumentation for characterization of P/MNPs composites.

In this work, [FeNi/Cu]<sub>4</sub>/FeNi/Cu/[FeNi/Cu]<sub>4</sub>/FeNi meander GMI elements were designed and tested aiming to develop a new instrumentation for characterization of polymer/magnetic nanoparticles composites. We have obtained EEW Ni-NP and created polymer composites and tested them using a GMI sensitive element.

Ni MNPs were prepared by EEW method.<sup>5</sup> The specific surface area of the produced Ni MNPs was determined by low temperature nitrogen adsorption using Micromeritics TriStar

3000. Phase composition was characterized by X-ray diffraction, XRD (Bruker D8 DISCOVER). Transmission electron microscopy (TEM) was performed using JEOL JEM2100 microscope, and FEI TECNAI G2 Polarmicroscope. Scanning electron microscopy (SEM) studies were done using JEOL JSM-7000F microscope.

Ni-filled composites were prepared based on the commercially available (Isomer, Dzerzhinsk, RF) acrylic copolymer of 95% of butyl methacrylate and 5% of methacrylic acid hereafter marked as BMK-5. Molecular weight of BMK-5 was  $3.1 \times 10^5$  (viscometry). First, the stock 25% solution of BMK-5 in ethyl acetate was prepared. Then, the certain volume of the stock BMK-5 solution was mixed with the appropriate amount of dry Ni MNPs. The precursor polymer suspension was cast onto the glass surface and left to dry off for 48 h. The dried composite films were placed in a vacuum to be dried further without substrates. Composites containing 90% of Ni MNPs by weight were prepared. Magnetic measurements of MNPs and selected composites were made using vibrating sample magnetometry (VSM).

[FeNi(100 nm)/Cu(3 nm)]<sub>4</sub>/FeNi(100 nm)/Cu(500 nm) [FeNi(100 nm)/Cu(3 nm)]<sub>4</sub>/FeNi(100 nm) meanders were fabricated by optical lithography on the basis of multilayers deposited by rf-sputtering.<sup>3,6</sup>

In order to obtain thick FeNi film (above 200 nm) and to avoid the appearance of the transition into a “transcritical” state nanostructuring can be applied by using thin Cu or Ti sub-layers.<sup>3,5</sup> Sputtering was done in a magnetic field of 100 Oe applied in transverse direction to the long axis of the strip. Meander sensor 1 (M1) consists of 200 μm wide 14 strips, meander sensor 2 (M2) consists of 300 μm wide 10 strips. The two meander sensors were used in this experiment, both had the same area of  $4 \times 8 \text{ mm}^2$ . The goal of this particular work was not to achieve as high as possible GMI value and GMI sensitivity (which is usually a characteristic of the rectangle GMI element is the shape of elongated stripe<sup>3,4</sup>) but to provide sufficient sensitivity for the GMI

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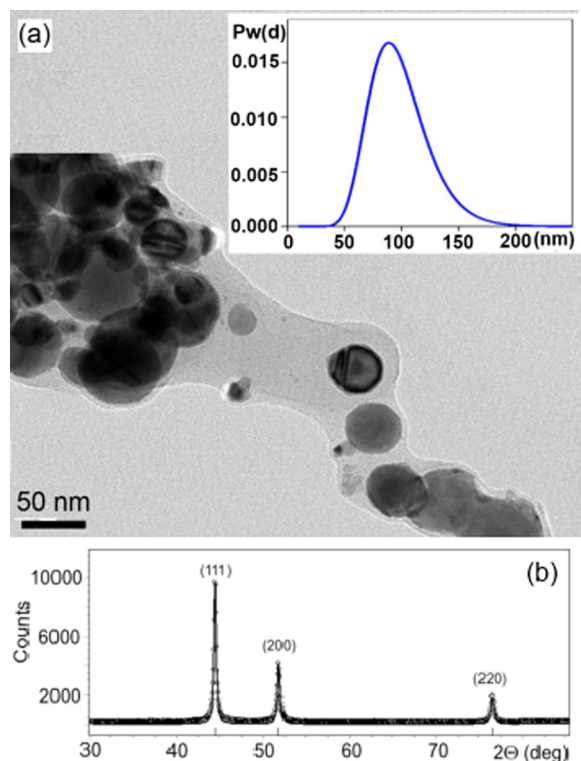


FIG. 1. (a) TEM image of 90% weight Ni MNPs in polymer. (b) XRD spectrum of Ni MNP. (Inset) Particle size distribution of Ni MNPs.

element with extended area, small dimensions, and geometry appropriate for studies of polymer coverings. Although different approaches are discussed in the literature<sup>7,8</sup> the possibility to improve GMI response by simple spread of thin polymer layer looks technologically attractive.

The absolute value of total impedance was measured as function of the applied magnetic field (up to 150 Oe) and frequency  $f$  (up to 150 MHz) both in increasing and decreasing magnetic fields in the configuration of “microstrip line” using radio frequency techniques.<sup>3</sup> The MI ratio  $\Delta Z/Z$  was defined with respect to a maximum external field  $H_{MAX} = 150$  Oe as follows:  $\Delta Z/Z(H) = 100 \times [Z(H) - Z(H_{MAX})]/Z(H_{MAX})$ .

Fig. 1 shows the summary of structural studies of MNPs and P/MNPs. The XRD spectrum contains the peaks typical for the intact metal Ni structure. XRD spectra were reasonably well fitted with the Rietveld method,<sup>5</sup> and crystallographic parameters were defined. The absence of NiO bands indicates that the passivating oxide layer at the surface is very thin (with the thickness below 5 nm). TEM and SEM images reveal that the Ni-MNPs are spherical in shape and non-agglomerated. The weighted average diameter ( $d_w$ ) of the spherical particles can easily be evaluated based on the specific surface area ( $S_{sp}$ ) of the MNPs:  $d_w = \frac{6}{\rho S_{sp}}$ , where  $\rho$  is the density of MNPs. Taken the Ni density of  $8.9 \text{ g/cm}^3$  and the value  $8.0 \text{ m}^2/\text{g}$  obtained for the specific surface nitrogen adsorption measurements, the average of 84 nm was calculated using the weighted particle size distribution ( $P_w$ )  $P_w(d_i) = \frac{m(d_i)}{\sum m(d_i)}$ . The obtained values of  $d_w$  for SEM (Fig. 1(b), dynamic light scattering, low temperature nitrogen absorption ( $85 \pm 5 \text{ nm}$ ) are consistent with each other and XRD data.

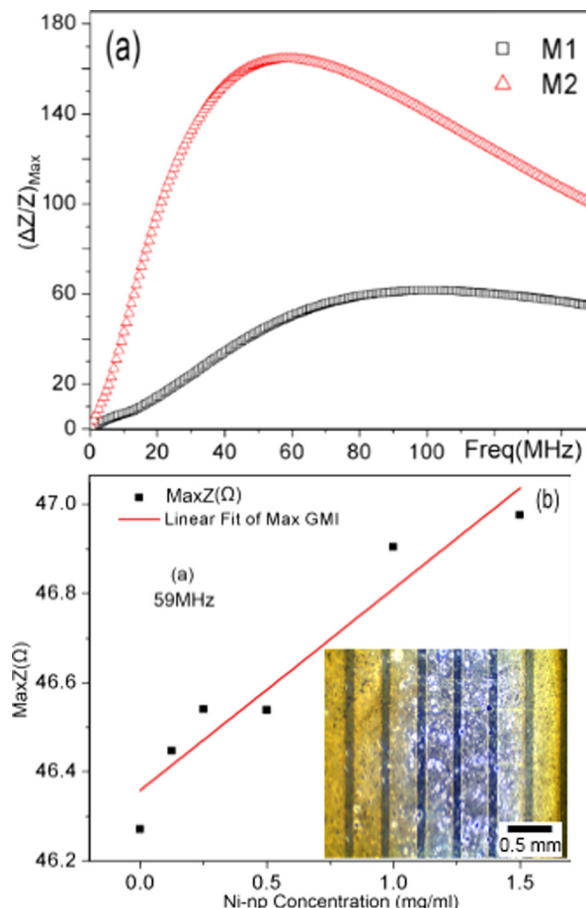


FIG. 2. (a) Frequency dependence GMI ratio of M1 and M2. (b) Maximum GMI dependence on increasing Ni MNPs concentration. (Inset) Meander sensor with Ni MNPs deposited on surface.

As the first step GMI of meander sensitive elements was studied for the samples with no covering or for the covering by thin layer of GE 7031 cryogenic varnish. The maximum value of impedance ratio  $(\Delta Z/Z)_{MAX}$  was 60% at 100 MHz for M1 and 165% at 60 MHz for M2 (Fig. 2) both for uncovered and GE varnish covered sensitive elements.

Pure MNP and P/MNP composite samples for GMI measurements were prepared by mixing the GE varnish and MNPs in a glass container with further treatment in an ultrasound cleaner to ensure proper mixing of the particles and their de-aggregated state. Next, using a micropipette  $20 \mu\text{l}$  of the mixture with MNPs or P/MNPs were deposited on the meander and dried for approximately 24 h allowing the solvent in the mixture to evaporate. For the preparation of the P/MNP sample, the total weight was increased to adjust for the extra weight of the 10% BMK-5 polymer, this was done to ensure the correct amount of MNP was used for each measurement. It must be noted that GMI measurements of the Ni MNPs in polymer are not influenced by the solvent in the solution, i.e., the BMK-5 polymer is only partially dissolved by the solvent leaving the MNPs in a de-aggregated state. The P/MNPs based composites deposited on the meander surface displayed a more uniform distribution of MNPs compared to the mixture with only pure MNPs which would agglomerate while the GE varnish was still drying.

The GMI measurements were made using MNP concentrations ranging from 0.0625 to 1.5 mg/ml. The relation



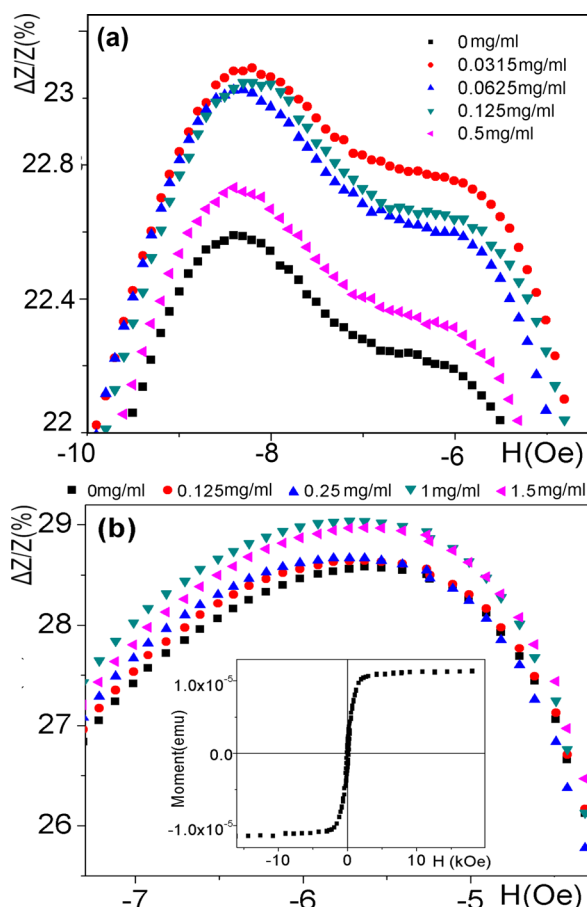


FIG. 3. M1(a) and M2 (b) magnetic field dependence of GMI ratio ( $\Delta Z/Z$ ) close-up at 100 MHz and 60 MHz, respectively. (c) Hysteresis loop of Ni MNPs in varnish per  $0.0009 \text{ mg/mm}^2$  corresponding to the  $1.5 \text{ mg/ml}$  concentration in GMI measurements.

between the increasing quantity of pure MNP and impedance is plotted for the M2 meander at 60 MHz (Fig. 2). The plot clearly shows a linear relationship with the concentration and impedance value. The origin of the sensitivity of the GMI of multilayered meander is similar to the sensitivity of giant magnetoresistance (GMR)<sup>10</sup> or GMI biosensors<sup>4</sup> in which the stray fields of the individual nanoparticles modify the external magnetic field and therefore the resistance or impedance of the detector. Although in a majority of previous studies related to magnetic biosensors, the stray fields were created by micro sized particles or nanoparticles in superparamagnetic state<sup>4,9</sup> it was shown recently that GMI sensitive element may have a capacity to detect different concentrations of carbon nanotubes.

The magnetic field dependence of the GMI ratio ( $\Delta Z/Z$ ) has been plotted for different concentrations for both the meanders (Fig. 3). The M1 meander was covered with 90% P/MNP and M2 with MNP. The different values of the concentrations plotted are due to the different contributions of the P/MNP and pure MNP. The P/MNP seems to increase the GMI ratio by 0.5% from 0.0315 mg to 0.125 mg of MNP, with further increase in particle content the GMI ratio decreases. The same GMI increase of 0.5% is found for the M2 measurements but with quantities of MNP of 1 mg and 1.5 mg. First of all, in all studied cases, we were able to detect measurable difference in GMI responses of meanders sensitive element with different kind of polymer coverings.

As in the case of carbon nanotubes<sup>10</sup> generally, non-linear dependence of the GMI ratio on the concentration was observed for whole concentration range under consideration. Second, we were able to detect quite small amount of the MNPs embedded in the covering ( $9 \times 10^9$  particles for a concentration of  $0.0325 \text{ mg/ml}$ ). Third, in certain concentration range, the GMI responses were increased by the presence of the P/MNPs coverings.

In order to understand the origin of the sensitivity of GMI meander element let us analyse the shapes of the hysteresis loops of the composites. Fig. 3(c) shows magnetic properties of MNPs in varnish for selected concentrations. That for the magnetic fields of the order of 6 Oe in which the GMI responses have the highest sensitivity to the concentration of the MNPs in the composite, these composites are characterized by rather high magnetization value (quite close to the saturation magnetization), i.e., indeed one can expect the influence of the fields created by the presence of the MNPs. For example, increases in GMI for certain concentrations could be explained by a reduction of stray fields due to the surface irregularities (closing the magnetic flux). Another important mechanism can be the following. The magnetic anisotropy of meander with opened magnetic flux is rather complex due to contributions of the shape anisotropy and interactions between the structural elements carrying the high frequency currents. The presence of polymer covering with MNPs dried on the surface of the meander can result in homogenization of the stray fields and changes in the magnetic dynamic permeability of the GMI element.

Ni nanoparticles were prepared by EEW method and characterized by different methods as well as polymer/Ni nanoparticles composites. GMI  $[\text{FeNi}(100 \text{ nm})/\text{Cu}(3 \text{ nm})]_4/\text{FeNi}(100 \text{ nm})/\text{Cu}(500 \text{ nm})[\text{FeNi}(100 \text{ nm})/\text{Cu}(3 \text{ nm})]_4/\text{FeNi}(100 \text{ nm})$  meanders were fabricated by optical lithography. The influence of MNPs on GMI responses of meander sensitive elements where studied. It has been shown that a small quantity of MNP or P/MNP can be detected by GMI when deposited on the meander surface. These results indicate a possibility of using GMI meander detector for characterization of polymer/magnetic nanoparticles composites.

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